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A comparison of the energy use of *in situ* product recovery techniques for the Acetone Butanol Ethanol fermentation

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Abstract

The productivity of the Acetone Butanol Ethanol (ABE) fermentation can be significantly increased by application of various *in situ* product recovery (ISPR) techniques. There are numerous technically viable processes, but it is not clear which is the most economically viable in practice. There is little available information about the energy requirements and economics of ISPR for the ABE fermentation. This work compares various ISPR techniques based on UniSim process simulations of the ABE fermentation. The simulations provide information on the process energy and separation efficiency, which is fed into an economic assessment. Perstraction was the only technique to reduce the energy demand below that of a batch process, by approximately 5%. Perstraction also had the highest profit increase over a batch process, by 175%. However, perstraction is an immature technology, so would need significant development before being integrated to an industrial process.

Keywords
In Situ Product Recovery, ABE Fermentation, Process Simulation, Energy, Economics

1 Introduction

Butanol is a commodity chemical that can be produced via the Acetone Butanol Ethanol (ABE) fermentation. The fermentation is limited by high product toxicity especially of butanol, therefore only reasonably dilute product concentrations (~20 gABE/L) are attained (Green, 2011). This affects the energy requirement for product separation and purification (Durre, 2008), which is important as the energy cost for the process was the second highest overall production cost, contributing 14%, behind that of the feedstock (79%) in a conventional batch ABE process (Green, 2011; Pfromm *et al.*, 2010). To overcome product toxicity, *in situ* product recovery (ISPR) has been applied and extensively researched for the ABE fermentation at laboratory scale (Abdehagh *et al.*, 2014). ISPR also provides the potential to increase the plant's production capacity from the same fermenter volume, through increased fermentation productivity and the use of fed-batch fermentations (Ezeji *et al.*, 2004a). While ISPR has been proven to be increase productivity and yield of the ABE fermentation, a definitive identification of the optimum technique has remained elusive.

A wide range of ISPR techniques have been investigated for compatibility with the ABE fermentation process. They can be compared using three key criteria. These are: the technique's ability to remove the product from the fermentation broth, the energy requirement and the economic impact. A meaningful comparison of the various techniques based on the extant literature is difficult for various reasons: differences in experimental method, ranging from media composition and microorganism strain to reactor configuration, for example. Only a few techniques have been subject to a

comprehensive energy and economic analysis. The most mature techniques for ISPR are gas stripping, pervaporation, vacuum (and flash) fermentations, liquid-liquid extraction (LLE), perstraction and adsorption, which have been highlighted in the recent review by Staggs and Nielsen (2015). All of these techniques have been proven to reduce product inhibition, but none are sufficiently developed for application to an industrial process (Van Hecke *et al.*, 2014).

The effects of application of the various modes of ISPR on the process have been widely studied, generally demonstrating improvements in productivity and the reduction of product toxicity (Ezeji *et al.*, 2010), whereas the associated energy demand of ISPR processes are often absent from literature (Van Hecke *et al.*, 2014). This paper focuses on the energy demand associated with ISPR techniques. A rough economic assessment of each process is made to compare the payback time associated with adding an ISPR technique to an existing batch plant.

It is known that ISPR can reduce downstream process energy demand, but the effect on the whole ABE production process has not been accounted for (Van Hecke *et al.*, 2014). Some limited energy analysis has been performed (see Table 1). A wide range of values have been calculated, based upon varying assumptions and process designs. The assumptions and process designs have been stated in Table 1's legend. These substantial differences between the various process designs and calculation methods make it extremely difficult to compare results across techniques.

In this work, comparative simulations have been performed for all ISPR techniques presented in Table 1. Simulations for perstraction were considered independently from liquid-liquid extraction, unlike that performed by Oudshoorn *et al.* (2009). The process

simulations allowed for comparison of both the effectiveness of the ISPR techniques, and the energy demand for both the ISPR technique and the downstream processing. The simulations were also beneficial for an economic assessment of the techniques.

2 Process Simulations

Process simulations were produced for a 50,000 te/yr ABE production plant with IPSR, using UniSim Design (Honeywell). A 50,000 te/yr plant was simulated as representational of a retrofit of a medium sized bioethanol plant for the production of biobutanol. The plants were compared on a production rate basis. This was based on a constant feed rate and a fixed conversion rate, accounting for the increased productivity of an ISPR fermentation, allowing for inefficiencies in the ISPR technique to be observed. The fermentation concentration was controlled to a maximum of 5g/L, in the stream returning to the fermenter after every ISPR technique. This is below the inhibitory concentration of butanol, allowing every ISPR process to have the same production rate hence the same fixed conversion rate. It is assumed that the ISPR techniques have no physiological impact on the bacteria. The thermodynamics of the process were described using the Extended-NRTL (Non-Random Two Liquid) model, based on previously being used for simulations of ABE and ethanol fermentations (Oudshoorn *et al.*, 2009; van der Merwe *et al.*, 2013; Wooley & Putsche, 1996). This model is good for multicomponent, azeotropic, dilute systems, like that experienced with the ABE fermentation. The Extended-NRTL version of the model is better suited to the wide temperature and concentration ranges present in the ISPR systems, utilising interaction parameters as a function of temperature. Two models were used; one with the binary coefficients estimated using UNIFAC vapour-liquid equilibrium, the other using UNIFAC liquid-liquid equilibrium. The model used depended on the mass

transfer occurring in each unit operation. Model applicability was confirmed by comparing it to experimental data from Stockhardt and Hull (1931), see supplementary materials Figure S1. Good correlation between the experimental data and model, especially for the low butanol concentrations (3-8g/L) when ISPR occurs with the average error between bubble and dew point temperatures being 0.59% and 0.75%, respectively. Additionally the model was compared to experimental, ternary ABE data (Perelygin V.M, 1980), where the model predicted the equilibrium concentrations with good accuracy. The average error in bubble point temperatures was 0.46%, and an average error of 11% for ABE concentrations.

The fermentations and downstream separations via distillation were based upon the work of Mariano *et al.* (2011a), using the same stoichiometric reactor model and downstream processing route. However, one alteration to the downstream processing route was made by adding the ISPR ABE-rich stream *after* the beer column rather than before. The beer column is used to concentrate the ABE in the fermentation and remove any unwanted components from the product stream, such as substrate, acetic acid and butyric acid which have not been converted to products, and biomass. For ISPR to have a positive impact the product recovery should be a concentrated ABE stream with no contaminants, therefore it will be not need to be pre-processed before ABE purification negating the need for it to be passed through the beer column. This agrees with Huang *et al.* (2014), with acetone being removed first, followed by ethanol then butanol and water. The beer column is still present in the simulations for processing the remaining fermentation broth at the end of the fermentation.

The reactor conversion was based on an ABE product ratio of 23:75:2 (wt%), this considers a process using a high-butanol-producing strain. This is similar to the ratios

seen by Tanaka *et al.* (2012). 80% of the product is produced via ISPR methods with a constant substrate concentration of 20g/L in the reactor. During the ISPR process the reactor conversion was set based on the amount of substrate converted to each product, ABE, or intermediate, acetic and butyric acid, based upon the stoichiometric reactions provided by Mariano *et al.* (2011a). The percentage of substrate converted into acetone, butanol, ethanol, butyric acid, acetic acid and biomass is 6.24%, 15.9%, 0.34%, 2.24%, 1.64%, 46.7%, respectively. Carbon dioxide and hydrogen are also produced during the fermentation; these are represented as by-products of these reactions. As the simulation is performed under steady-state conditions the conversions ensure that there is a uniform concentration of substrate, products and biomass. For this reason, the fermentation broth is not returned to the fermenter after ISPR has been applied; in real fermentations the fermentation broth will be returned to the fermenter for further processing.

The reactor was assumed to be continuous. Whilst this is not a direct representation of the ABE process, which would typically be a batch or fed-batch fermentation, it allows for a direct comparison of the energy demand for the ABE production process with different ISPR methods. Other reactor types such as biofilm or immobilised reactors have been demonstrated at laboratory scale but have not yet been demonstrated commercially for the ABE fermentation therefore have not been considered for simulation (van der Merwe *et al.*, 2013). An additional continuous reactor was added to the process to represent the batch culture produced at the end of the fermentation. For the batch process the reactor conversion based on substrate consumption for acetone, butanol, ethanol, and biomass production was 14.2%, 36.2%, 0.78% and 48.9%, respectively. It was assumed that no acids would be present at the end of the batch

phase, as they would have been re-assimilated into ABE to achieve the maximum yield possible. Hence, the whole process energy could be calculated, allowing comparison to standard batch fermentations.

The ISPR techniques simulated were flash fermentation, vacuum fermentation, gas stripping, pervaporation, liquid-liquid extraction, perstraction and adsorption. These methods of ISPR were selected as they are the most developed techniques and performance information is available in conjunction with the ABE fermentation. Key process conditions were based on experimental data from literature. The process flow diagrams used for the simulations in UniSim Design are shown in Table 2 and Figure 1, along with the specific details relating to each ISPR technique.

In addition to simulating the ABE fermentation with ISPR a conventional batch ABE fermentation was also simulated. As the application of ISPR increases the productivity of the plant, hence annual production, the batch plant capacity was assumed to be 18,000 te/yr. This is representational of the same capacity plant, without the ISPR technique applied. The annual production is reduced due to batch fermentations, but the annual number of fermentations per year is increased due to shorter fermentation times, therefore accounting for a 63% decrease in production capacity.

Van Hecke *et al.* (2014) suggested that there are limitations to current simulation software with regards to its ability to incorporate ISPR technologies into the simulations. The limitations, such as the inability to effectively simulate a membrane or adsorption process, stem from the software being designed for the traditional petrochemical industry. Perhaps this explains the lack of information surrounding process energy consumption. As UniSim Design was designed for the chemical industry

it is not wholly suited to simulating a bioprocess, although it is ideal for simulating separation processes of ABE from water. To overcome the limitations of the software, the following assumptions were made:

- Stoichiometric, continuous, steady-state simulation of the fermentation. The continuous stream from the fermenter was assumed to represent the desired conditions in the reactor. The flow leaving the reactor is representation of the conditions and concentrations that would be observed during a fed-batch, ISPR fermentation
- The NREL database was used to provide properties for biomass (Wooley & Putsche, 1996). The chemical composition of biomass was defined as $CH_{1.57}N_{0.23}O_{0.39}S_{0.0035}$ allowing the molecular weight to be calculated as 24.6 kg/kmole (Wooley & Putsche, 1996). This allowed for creation of a biomass component the same as that used by Mariano *et al.* (2011a)
- Acetic acid and butyric acid were produced as reaction intermediates. They were assumed to be present in the broth in their dissociated form, as the pH of the broth (minimum pH 5 (Qureshi & Maddox, 2005)) is greater than the pKa of the acids (4.76-4.88). This reduces acid removal from fermentation broth during ISPR, particularly during evaporative techniques, to ensure no contamination of the final product
- The rate of product formation is assumed to be equivalent to that observed at low solvent titres, ~5 gABE/L, this concentration is considered non-inhibitory to the fermentation (Ezeji *et al.*, 2004a)
- Membranes can be simulated as component splitters, using membrane flux data (from literature, Table 2) to calculate retentate and permeate component

fractions. The pressure drop across the membrane was not simulated, though the pressures are controlled in the streams leaving the component splitter. This is particularly important for pervaporation

- The extractant oleyl alcohol, for LLE and perstraction, is simulated as a hypothetical component, using UNIFAC to calculate component properties such as molecular weight, critical temperature, critical pressure, critical volume and acentricity. UNIFAC was used as no experimental data was available for these values; experimental data would have provided greater accuracy
- Adsorption and desorption were simulated as conversion reactions, with carbon representing the adsorbent. As the process is simulated as a series of conversion reactions carbon forms an ideal basis for matrix, as it is present within UniSim's component database and activated carbon has been used as an adsorbent for ABE (Groot & Luyben, 1986)
- Evaporation-based techniques utilised a gas scrubber to reduce the ABE concentrations in the gas emission streams to capture product and ensure the plant's gas emission were within workplace exposure limits. The limits were acetone, 500ppm, butanol, 50ppm, and ethanol, 1000ppm, based upon United States Department of Labor Occupational Safety and Health Administration (OSHA, 2014)
- Final product concentrations for acetone, butanol, ethanol and water were 99.5wt%, 99.5wt%, >80wt% and 99.5wt% respectively
- No losses of product in the downstream distillation process.

Further to this, no energy integration was applied to the simulations, to allow for an equal comparison of maximum energy demand across each technique. Using process

stream information from the simulations grand composite curves were developed for each ISPR technique, to understand if any energy can be saved from heat integration.

2.1 Economic Analysis Method

An economic analysis was performed based on the results of the simulations. The fixed capital costs were estimated using an updated Bridgewater's method, described in Towler and Sinnott (2013). The variable operating costs were based upon the mass balance and relative energy requirements extracted from the process simulations. For the economic analysis the product yield was assumed to be 0.32 kg ABE/kg substrate, 0.1 kg biomass/kg substrate and 0.58 kg gas/kg substrate. The feedstock and product values were taken from Kumar *et al.* (2012) and Qureshi *et al.* (2013). The process simulations were developed to represent a retrofit of a medium sized bioethanol plant. The economic analysis considers the impact of adding ISPR to the plant, by calculating the additional fixed capital required, the additional profit made and associated payback times due to the addition of ISPR.

3 Results and Discussion

The process simulations were designed to assess the process energy demand for ABE fermentations incorporating ISPR. The separation efficiency was used to compare how well each ISPR technique can remove ABE from the fermentation broth. The maximum energy demand and separation efficiencies are shown in Figures 2 and 3, respectively. Figure 2 shows the upstream and downstream contributions to the total energy, where the upstream is in relation to the fermentation and ISPR process shown in Table 2 and downstream is the distillation purification process shown in Figure 1.

The simulation results provide insight into both the energy required for the fermentation and ISPR technique, and the impact the ISPR method has on the downstream energy consumption. All energy uses were included in the analysis, including the energy required for heating, cooling and electrical pump duty. This is in contrast to previous work, such as Mariano *et al.* (2011a) and Mariano *et al.* (2012), where only the energy for the vacuum pumps is considered. A sensitivity analysis was performed to assess the reliability of the simulations and the assumptions on which they were based. The variables tested and their ranges are shown in Table 2.

3.1 Upstream Energy

Figure 2 shows a wide range of energy demands for upstream section of the process. Of all the ISPR techniques, perstraction has the lowest upstream requirement, and LLE the highest. The high LLE energy demand is due to the large volume of extractant used for removal of the butanol from the fermentation broth. More extractant means more energy is required for butanol removal via distillation and sterilisation of the extractant before coming in contact with the fermentation broth. The recovery of ABE from any separating agent and renewal of the separating agent has been included as part of the upstream energy demand. For LLE this makes a significant contribution to its high energy demand. Oleyl alcohol is also used as the extractant in the perstraction process, but, as a membrane separates the fermentation broth and extractant phases, smaller volumes of oleyl alcohol are required, less energy is required for renewal of the extractant and no energy is required for dispersion and coalescence of the two phases.

3.2 Evaporative Techniques

In general, evaporative techniques have a high upstream energy demand. This is largely due to the low concentration of ABE in the recovered stream and the associated

difficulties of capturing the gas from this stream. For example in gas stripping, the ABE is heavily diluted as the gas flow rate required to maintain the butanol concentration in the fermentation broth below inhibitory levels is substantial. In these simulations the butanol concentration in the bioreactor was maintained at 3.5g/L, based on the concentrations seen by Ezeji *et al.* (2004a). Water is also removed from the fermentation broth with evaporative techniques, further diluting the vapour stream. Energy was then required to capture the ABE from the vapour phase. It is difficult to capture 100% of the solvents at 1-2°C due to the low vapour pressures of the ABE, in particular acetone, which has a vapour pressure of 0kPa at approximately -50°C. This was also remarked upon by Ezeji *et al.* (2004a) in their laboratory-scale trials. It was not possible to capture the entire product from the vapour phase within the temperature ranges for condensers seen in literature. Clearly, final product titres, therefore yields and productivities, for evaporative-based experimental techniques will contain substantial errors. This is why the evaporative techniques data have larger error bars in Figures 2 and 3.

Pervaporation has the smallest evaporative downstream energy. The separation is largely achieved by a non-porous membrane. This reduces the amount of water being removed from the fermentation broth. Less energy will be required for evaporation and condensation as less water is being removed from the fermentation broth. For all other evaporative techniques there is direct contact between the liquid and vapour phase.

The flash and vacuum techniques are very similar processes, but they exhibit different upstream energy requirements due to the location of the separation. Flash separation is performed in an external vessel, whereas vacuum separation takes place inside the fermenter. The application of a vacuum to the fermenter is likely to have an additive

effect to stripping of the ABE by the gasses produced in the fermentation. Mariano *et al.* (2011b) came to this conclusion when comparing vacuum and flash separations. This also goes some way to explain the differences seen in the separation efficiencies between the two techniques, Figure 3. The results show that vacuum fermentation is superior to flash separation, but the cost of implementing this needs to be considered particularly when considering the size of the fermentation vessel that needs to withstand 6.5kPa vacuum.

The evaporative techniques are more sensitive to changes in the simulations. This is probably due to the amount of solvent removed from the broth being a direct result of the energy applied, unlike other techniques which use a separating agent to remove the product from the fermentation broth. All changes to the process occurred in the upstream section, but significant variance is seen in the downstream process, particularly with gas stripping.

3.3 Downstream Separation

In contrast to the upstream energy demand, LLE has the lowest downstream energy requirements for ISPR techniques. This low energy requirement means that, overall LLE is not the most energy intensive ISPR technique. Instead this is gas stripping, which has one of the highest upstream energy requirements and the highest downstream energy requirement. The downstream energy is almost identical to the energy required for upstream processing.

The four largest ISPR downstream energy demands are for the evaporative separation techniques. Due to the large amounts of water removed from the fermentation broth during the *in situ* recovery step. The downstream energy requirement is heavily dependent on the concentration of ABE entering the downstream system. The ISPR

techniques with the lowest downstream energy demands have much higher concentrations of ABE at this point. LLE produces the most concentrated stream from *in situ* recovery. It is therefore not surprising that LLE and perstraction have the lowest downstream processing energy demands, as they both utilise a non-polar organic extractant for separation. The extractant's affinity for water is extremely low, hence the high product concentration and low downstream energy demand.

Compared to a standard conventional batch ABE fermentation, it can be seen that all ISPR processes have a greater upstream energy, Figure 2. This is to be expected as at least one additional unit operation is being added to the upstream process. In contrast, all ISPR techniques, other than gas stripping, have a lower downstream energy than the standard batch process. This is expected due to the increased concentration of ABE in the downstream section of the process, resulting in lower downstream energy demand. Interestingly, however, when the energy demand for the whole process is considered, only perstraction has a lower total energy than the batch process. The reduction is nearly 5%.

3.4 Separation Efficiency

From the simulations it was also possible to quantify a comparative degree of recovery. This is not evident in the existing literature. The separation efficiency is a comparison of the amount of ABE separated from the fermentation broth during ISPR compared to the amount of ABE present in the fermentation. As all ISPR simulations had the same feed flow rate and reaction scheme, an assessment of the techniques' abilities to remove ABE from the fermentation broth can be made (see Figure 3). The simulation results show that the highest separation efficiency is achieved by LLE, then by vacuum fermentation. Based on the simulations the minimum separation efficiency required to achieve a maximum fermentation broth concentration of 5g ABE/L is 36%. This

minimum threshold is achieved by all techniques other than flash separations and gas stripping. This is due to the difficulty of capturing the ABE from the dilute vapour phase generated as part of the ISPR method. Adsorption has the potential to be a very selective technique for the recovery of ABE from the fermentation broth. The downside is that product recovery and adsorbent regeneration both require steam. The steam is a means of direct heat application for separation, although some water vapour will be transferred into the product recovery stream to downstream processing. Other methods of adsorbent regeneration are possible, such as chemical recovery, for example with methanol, but the ABE would then need to be recovered from the methanol, further increasing the number of process steps (Yang *et al.*, 1994).

3.5 Membrane Technologies

Based on the results shown in Figures 2 and 3, the effect of using a membrane technology can be observed. In the case of LLE and perstraction, it can be seen that the membrane option (perstraction) has a lower degree of recovery and energy demand for upstream processing although it has a slightly higher downstream energy demand. The comparison for evaporative technologies is slightly more difficult, as both flash separation and vacuum separation are equivalent to pervaporation. As vacuum separation is a significantly more effective technique than flash separation this will be used here as the comparison with pervaporation. Pervaporation has a lower degree of recovery and lower energy demand than vacuum separation. The energy differences between vacuum separation and pervaporation are small compared to those seen for the extractive processes.

From these simulation results it is clear that the use of membranes currently hinders the effectiveness of the separation, as both membrane techniques have a lower separation

efficiency compared to vacuum fermentations and LLE. It is also worth noting that with current membrane technology the mass transfer differences across the membrane, between pervaporation and perstraction, do not have a large impact on the separation efficiency. The relatively large error associated with the separation efficiency of pervaporation and perstraction was expected due to the software's inability to directly simulate a membrane operation; therefore there was greater reliance on published experimental data, and there is very limited information available in relation to the membrane permeability and diffusion rates, particularly for perstraction.

In terms of energy of the process, the use of membranes reduces the energy demand of the process although it is questionable if the energy reduction for evaporative techniques is enough to justify pervaporation over vacuum fermentation. Vacuum fermentations and pervaporation both require 87MJ/kg ABE for the upstream separation and the downstream separation for pervaporation is 3MJ/kg ABE less than that required for vacuum fermentations. The greater separation efficiency corresponds to an increase in ABE recovered, which is likely to generate a bigger profit then any savings made from reducing the energy by 3MJ/kg ABE. These results can probably be improved through membrane development and optimisation to make membrane techniques more widely used.

3.6 Heat Integration

Heat integration can be used to reduce the energy demand of the process. González-Bravo *et al.* (2016) have previously investigated heat integration for downstream biobutanol separation, assessing two distillation systems and two hybrid liquid-liquid extraction/distillation systems. Their results indicated that the energy demand can be reduced through heat integration but they did not consider applying it alongside ISPR.

Using process stream information from the simulations it was possible to assess the potential for reducing the heating and cooling demand. Grand composite curves were developed for each simulation, so the minimum energy for heating and cooling could be calculated, using a ΔT_{min} of 20°C. The calculated minimum energy does not include energy for electrical equipment such as pumps and compressors or energy required to maintain a constant temperature in the bioreactor or other equipment (for example membrane units).

The minimum heating and cooling energy required for a batch process was 19.7 MJ/kg ABE, this was similar to that required for gas stripping (18.3 MJ/kg ABE) and flash separation (18.9 MJ/kg ABE). These ISPR techniques did not achieve the minimum separation efficiency of 36%, therefore the concentrations of ABE in the downstream separation section are going to be equivalent to that of a batch system so similar minimum energies is not unexpected.

The techniques which achieved a separation efficiency greater than 36% all had significantly lower minimum energies for heating and cooling, apart from liquid-liquid extraction with a minimum energy of 54.3 MJ/kg ABE. This was the largest minimum energy of all the techniques, and is largely related to the energy required to recover the ABE from the extractant and subsequently cool the extractant for reuse. Vacuum fermentation, pervaporation, adsorption and perstraction all have similar minimum heating and cooling energies of 9.5 MJ/kg ABE, 6.3 MJ/kg ABE, 5.5 MJ/kg ABE and 4.1 MJ/k g ABE, respectively. These minimum energies are in the same order as the maximum total energy required in Figure 2. Having lower heating and cooling requirements means that heat integration can be applied to the process and this will further reduce the process energy demand. Applying heat integration would increase the

capital cost due to heat exchanger requirements. Further work would be required to fully understand the possible heat exchange networks and to optimise the energy savings that could be made, alongside the additional capital cost for installation of the heat exchange network.

3.7 Literature Comparisons

It is difficult to make comparisons between the simulation results presented here and previously published results in the literature. As previously stated, the results from literature in Table 1 rely on assumptions that are different in each source and vary from some of the assumptions used in these simulations. The major difference with this work is that the data presented in Table 1 are primarily concerned with the energy required for just the separation technique, rather than the entire upstream and downstream process. The biggest difference seen between the literature results and this is the energy requirements for LLE. The overall energy, 185MJ/kg ABE (Figure 2), was one of the highest where as in previous studies the energy demand for LLE (7-9MJ/kg BuOH) was one of the lowest (Table 1). It is suspected that the previous studies did not include the energy required to separate the ABE from the extractant (Groot *et al.*, 1992; Oudshoorn *et al.*, 2009; Qureshi *et al.*, 2005). This difference can have a significant impact on the viability of the process from an energy demand assessment.

In contrast, other authors such as Salemme *et al.* (2016) have only considered alternative downstream processing routes, replacing the traditional batch column with either a gas stripper or LLE unit using 2-ethyl-1-octanol. The results provided by Salemme *et al.* (2016) include heat integration, therefore could be compared to these minimum energy demand results presented in this work. For gas stripping they achieved a specific energy requirement of 15.3 MJ/kg, this is very similar to the minimum energy

demand of 18.3 MJ/kg ABE achieved in this work; indicating that that gas stripping is very similar to a distillation column. The results for LLE vary greatly with Salemme *et al.* (2016) achieving 9.9 MJ/kg whereas this work presents a minimum energy requirement of 54.3 MJ/kg ABE. It must be noted that this paper has an additional distillation column and the primary separation is occurring at concentrations less than 10g/L whereas the feed concentration used by Salemme *et al.* (2016) is 30g/L. This difference in concentration can have a large impact on the energy required for separation as demonstrated by Mariano *et al.* (2011a).

3.8 ISPR versus Batch Processing

It is well-documented that for the traditional ABE fermentation process the energy-intensive nature of solvent purification by distillation is one of the biggest challenges to be overcome (Dürre, 2007; Ezeji *et al.*, 2004b; Green, 2011). The information that is available tends to compare the energy for ISPR with other ISPR techniques, as shown by Table 1, with hardly any comparison to the conventional batch process, particularly in terms of the whole process. One of the significant results is that whilst the downstream energy requirement can be reduced through the application of an ISPR technique, increases in whole process energy are usually observed. This is confirmed by Mariano *et al.* (2012), in which the energy for a vacuum fermentation is considered. The same result was observed here (Figure 2) for all ISPR techniques, apart from perstraction, the whole process energy is greater than that for the batch process. This means that whilst the use of ISPR can reduce the downstream energy, it shifts the associated costs to an alternative part of the process.

It is clear that, generally speaking, non-evaporative processes will provide less intensive recovery and a greater annual production because of the high condensing power

required for acetone capture. The target output for the simulations was 50,000 tonnes ABE/year. In no ISPR technique was the entire product produced recovered, indicating that no technique is 100% efficient for recovery. As the substrate inputs were constant across every ISPR technique, the losses were able to be used to assess the effectiveness of each technique through the separation efficiency, Figure 3. The losses are due to the lack of development, in terms of scale up and optimisation, with the application of ISPR to the ABE fermentation. The only technique to have shown any systematic process development is LLE, which the simulations show has the highest separation efficiency (Roffler *et al.*, 1987; Roffler *et al.*, 1988). These losses need to be assessed for each technique. Minimisation of these losses will see an increase in product recovery, and a reduction in the energy requirement.

The results in Figure 3 show that the more developed techniques, vacuum fermentation and LLE, exhibit some of the highest recoveries of ABE from the fermentation broth. However, this is probably a result of their higher technology readiness levels than a fundamental phenomenon. Ideally, the results would have shown that the technique with the lowest energy demand also had the highest separation efficiency clearly indicating the best ISPR technique. As this is not the case, it means that a compromise is required when deciding on an appropriate technique. From the results presented here, perstraction appears to be the most favourable technique, as it is the only ISPR technique that gives an improvement over the batch process in terms of overall energy demand. Furthermore, it has the third highest separation efficiency, indicating good separation characteristics. Another possible advantage is that it is at a relatively early stage of development, so could yet prove to be more effective, if membrane technology advances or if economies of scale are realised for this technology.

Information concerning the development of these techniques from laboratory scale upwards is limited (Van Hecke *et al.*, 2014). The simulations are only a representation of the process, and advances in these technologies could significantly alter the results presented here. Improvements to the process could come in the form of membrane, adsorbent or extractant development. Development of these technologies at larger scales, thereby providing scale-up data would have a significant impact on the ABE process, particularly for adsorption and perstraction.

3.9 Economic Assessment

An economic comparison of the application of multiple ISPR techniques to the ABE fermentation has not previously been described in literature (Abdehagh *et al.*, 2014). This is due to the early stage of development of most ISPR techniques and scarcity of information about associated costs, such as membranes and adsorbents. An economic assessment has been completed by Roffler *et al.* (1987) for LLE using oleyl alcohol, compared to a batch plant to produce a the same quantity of butanol, indicating that 20% less capital was required due to reduced broth volume required and higher fermentation productivity. Abdi *et al.* (2016) have provided a comparison of a non-integrated ABE fermentation with an ABE fermentation integrated with flash separation at a pressure of 7kPa in an external vessel. This analysis demonstrated how the application of ISPR could significantly increase the profitability of the process, meaning the ABE fermentation would be more able to cope with fluctuations in the market price of butanol. van der Merwe *et al.* (2013) primarily focused on varying the downstream processing route, but found the application of ISPR, using gas stripping, with LLE as the first downstream processing step to be profitable. This is similar to the economic assessment have been performed by Liu *et al.* (2004), which considered the use of LLE

or gas stripping replacing the initial beer column, with varying distillation routes. Results shown in Table 3 compare the extra capital required and extra profit generated by utilising ISPR.

The upstream fixed capital required for each ISPR technique was based on the number of functional units in the process, for a plant in the U.S. Gulf Coast, January 2010. The downstream process capital expenditure was not considered as it is identical for every technique, due to having the same process configuration (Figure 1). This method agrees with other literature values for both a batch ABE plant and a plant with ISPR. Kumar *et al.* (2012) calculated that the capital cost for a batch plant producing 10,000 tonnes of butanol per year from corn to be $10.3 million. Nilsson *et al.* (2014) estimated the capital cost for an ABE plant with gas stripping to be approximately $62 million. Both values are similar to the results shown in Table 3, indicating a good estimate of the capital cost.

The operating costs were based on the annual production and the relative energy requirements (MJ/kg ABE) from the process simulations for the whole plant. The yield for the ISPR techniques has been assumed to be equivalent to a batch process, therefore each ISPR process has the same annual production and feedstock consumption. In reality the overall annual production would vary for each technique due to different efficiencies of the recovery; however for optimal economic efficiency all products need to be recovered at some point during the process, allowing the overall process yield to match the bacterial yield. The payback time for the addition of ISPR to an existing ABE fermentation plant was calculated and shown in Table 3. This compares the extra capital cost required for ISPR and the extra profit generated through the use of an ISPR technique. This method agrees with the method used by Abdi *et al.* (2016) who

observed operating costs for the integrated fermentation increase by 181%, this is similar to the results presented here where by the operating costs increased by 182% for vacuum separation and 205% for flash separation.

Similar to the results in existing literature, the use of ISPR increases the profitability of a fixed volume plant. In terms of comparing ISPR techniques, perstraction and adsorption have the joint lowest payback time, closely followed by vacuum fermentation. These three options also have the greatest increase in profitability over a batch process. The major variable affecting the profitability is the energy cost, which was determined from the process simulations. Thus is unsurprising that the techniques with the lowest energy demand also had the shortest payback time.

All ISPR techniques assessed could increase plant profitability (Table 3) through increasing capacity. The ISPR-based plants produce 2.75 times more product than that of the batch plant. Perstraction is the only ISPR technique to produce an equivalent increase in the profit. Gas stripping was the least profitable of all the ISPR techniques, only increasing the batch profit by 110%. This is due to the high energy demand of the process, (Figure 2), which, when combined with the increased capital cost, increases the additional payback time of the plant. The application of flash fermentation to the ABE process has a similar effect.

The economics of the process needs to be considered alongside the separation efficiency. Vacuum fermentation and LLE have the greatest separation efficiencies but fall in the middle of the range in terms of profit increase. These are also the most developed techniques, therefore have limited scope for development. Perstraction, adsorption and pervaporation have the greatest increase in profitability, but there is a

much greater scope for development to improve the separation efficiencies of the ISPR techniques; making the techniques more attractive for commercial ABE production.

The potential reduction in energy has been one of the driving forces for research into the application of ISPR to the ABE process. Perstraction is the only technique which has an energy demand lower than that of a batch process. It is a very similar technique to LLE and should be able to overcome some of the problems that occur when using LLE, specifically extractant toxicity and the volume of extractant required. The biggest difference between LLE and perstraction is the amount of product recovered, (Figure 3). Increasing the separation efficiency will help to make perstraction an ideal ISPR technique for the ABE fermentation. However, it has one significant disadvantage: its relative lack of development. Currently, perstraction exhibits low extractant rates, as there has been no systematic optimisation of the extractant and membrane materials vs. flow rates (Qureshi & Maddox, 2005). Significant membrane development occurred for the use of pervaporation in ABE pervaporation between Groot *et al.* (1984)'s use of silicone tubing and Van Hecke *et al.* (2012)'s use of commercially available pervaporation modules from Pervatech. If similar developments can occur for perstraction, it could prove to be the most cost-effective ISPR technique for the ABE fermentation.

4 Conclusions

Previously, no ISPR technique has been identified as being the best for removal of ABE from fermentation broth. This study shows that ISPR generally *increases* the overall energy requirement of the plant. The one exception found in this study is perstraction, which had the lowest overall energy requirement, leading to a 175% increase in profit

over a conventional batch plant. However, perstraction does not provide the greatest separation efficiency. This was achieved by LLE, which is a more mature technique. Through developments in perstraction membrane technology it could be possible to recover more ABE, matching the separation efficiency of LLE.

5 Acknowledgements

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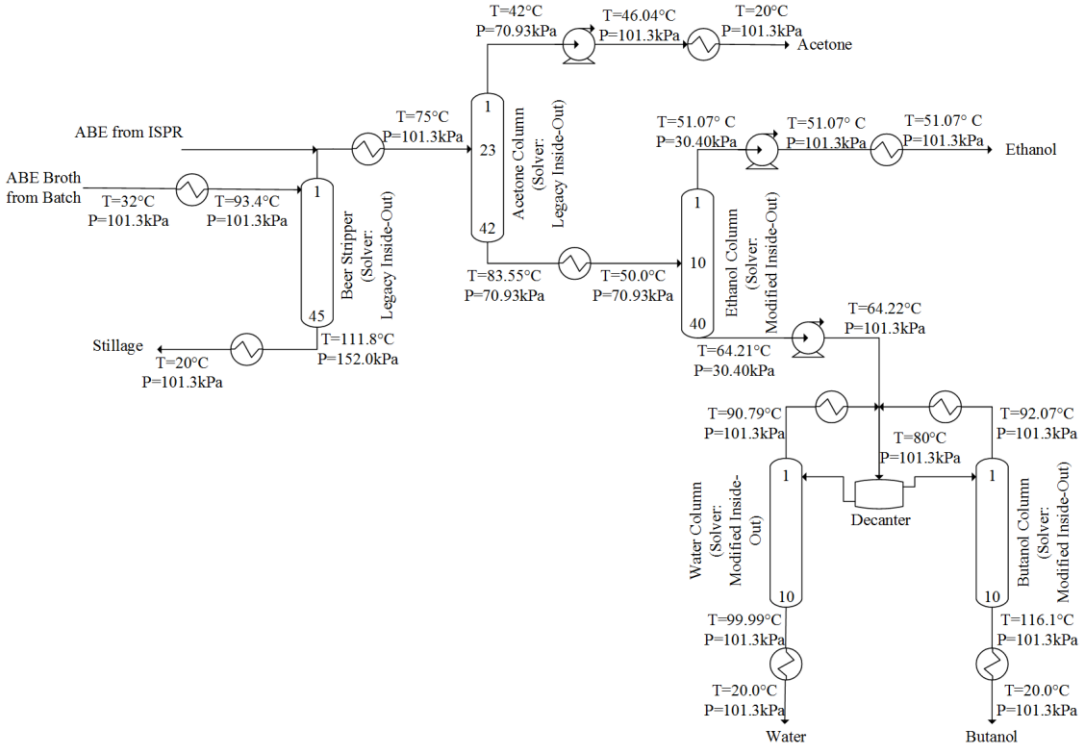


Figure 1: Downstream distillation stream conditions for simulation

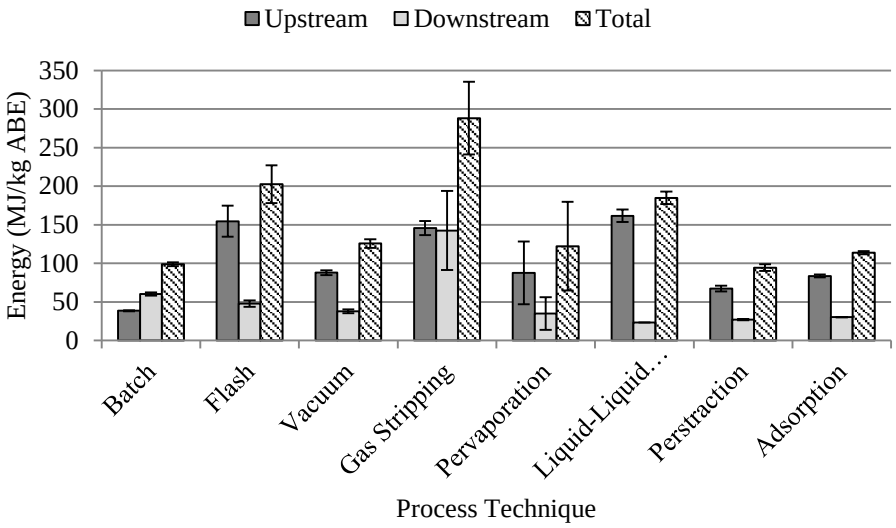


Figure 2: Maximum energy requirement for seven ISPR techniques (production rate 50kt/yr), representing individual upstream, downstream and total requirements, compared to a batch process (production rate 18 kt/yr).

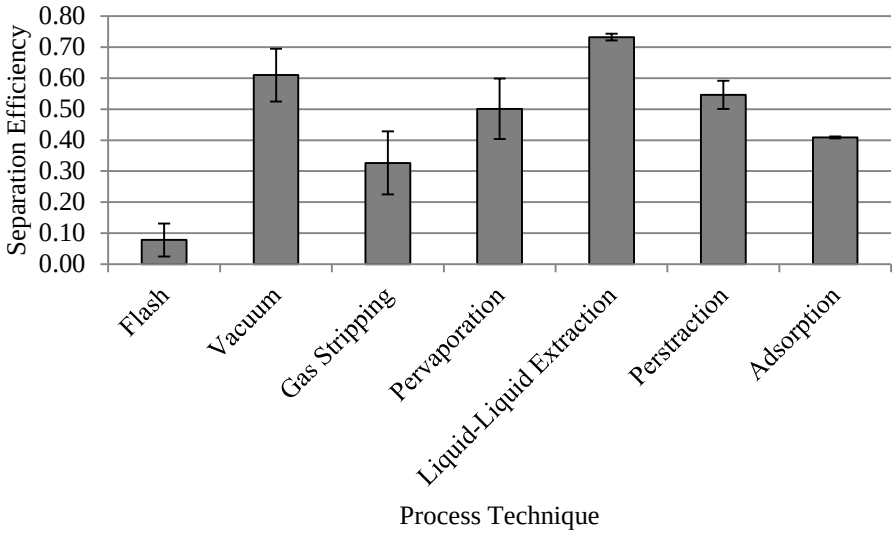


Figure 3: Separation efficiency for each ISPR technique

Table 1: Comparison of energy information in literature for ISPR

Technique	Oudshoorn *et al.* (2009)[a]	Qureshi *et al.* (2005)[b]	Groot *et al.* (1992)[c]	Nielsen and Prather (2009)[d]	Mariano *et al.* (2011a)[e]	Mariano *et al.* (2012)[e]	Salemme et al. (2016)[f]
	(MJ/kg BuOH)	(MJ/kg BuOH)	(MJ/kg ABE)	(MJ/kg BuOH)	(MJ/kg BuOH)	(MJ/kg BuOH)	
Flash					4.4-6.5		
Vacuum						10.2-15.6	
Gas Stripping	14-31	22	21				15.3
Pervaporation	2-145	14	9				
Liquid-liquid Extraction	7.7	9	14				9.9
Perstraction	7.7						
Adsorption	1.3-33	8	33	7.8			

[a] Energy required for separation of butanol from water. Calculations on steady state flow and enthalpy changes in the system. Using thermodynamic data from NRTL property package in Aspen Plus 12.1 (Oudshoorn *et al.*, 2009).

[b] Energy requirement for butanol separation from fermentation broth. Separation of acetone and ethanol is not considered. Unknown calculation method.

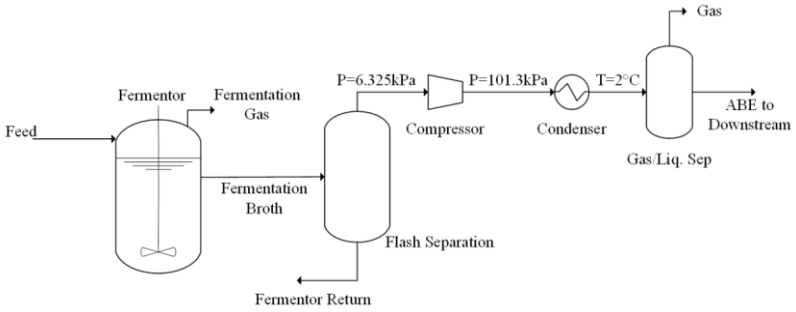
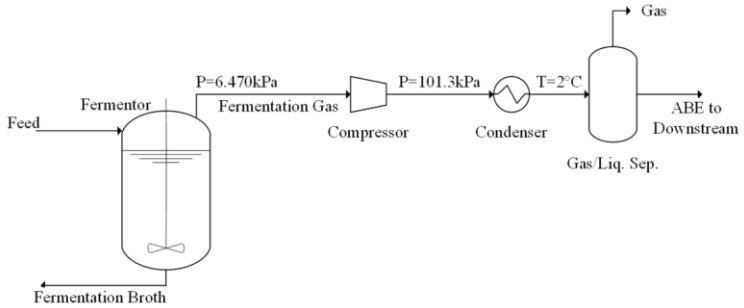
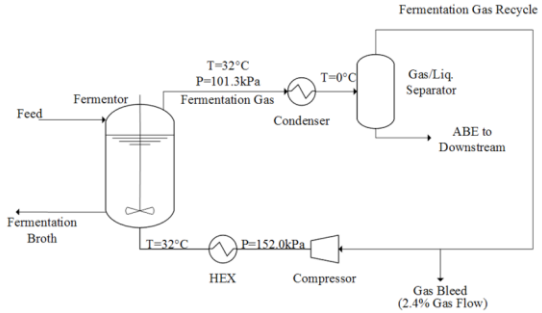
[c] Estimated total heat of recovery for the overall heat process. Based upon recovering by a two column system. A complete separation of acetone and ethanol is not considered. Unknown calculation method.

[d] Energy for adsorption and desorption process only. Calculated from mass balance.

[e] Electrical energy only for in situ separation, based on process simulations.

[f] Energy requirement for separation technique and a four column distillation set up. The proposed separation technique replaces beer column, not used for ISPR. All energy was made homogenous by expressing as fuel equivalents. No units were given by Salemme *et al.* (2016), but the specific energy demand for butanol is calculated as the total energy rate supplied divided by the mass flowrate of butanol in the system and the lower heating value of butanol.

Table 2: Description of ISPR technology simulations

ISPR Technique	Diagram	Sensitivity Analysis Changes	
		Variable	Range
Flash Fermentation (Mariano *et al.*, 2008)		Vacuum Pressure (kPa) Condenser Temperature (°C)	0.5925-6.9575 0-4
Vacuum Fermentation (Mariano *et al.*, 2011b)		Vacuum Pressure (kPa) Condenser Temperature (°C)	5.823-7.117 0-4
Gas Stripping (Ezeji *et al.*, 2004a; Ezeji *et al.*, 2003)		Compressor Pressure (kPa) Condenser Temperature (°C) Gas Bleed	101-202 -2 - 2 2.4-12.2%

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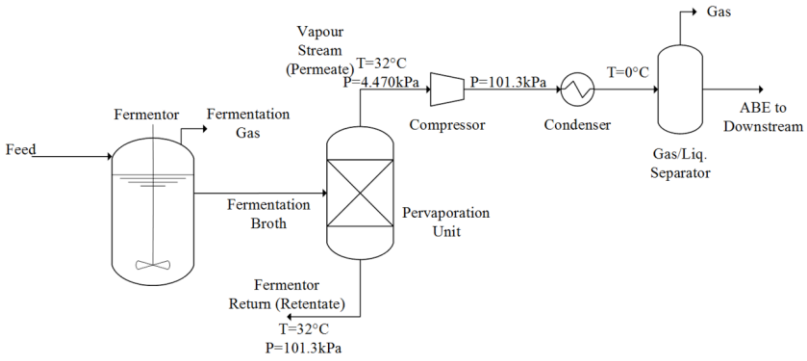
<tr>
<td>Pervaporation (Wu et al., 2012)</td>
<td>


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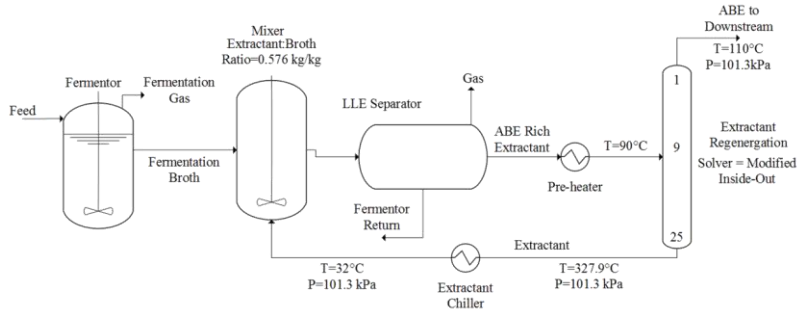
<tr><th>Component</th><th>Acetone</th><th>Butanol</th><th>Ethanol</th><th>Butyric Acid</th><th>Acetic Acid</th><th>Dextrose</th><th>Biomass</th><th>Water</th></tr>
<tr><td>Permeate Split</td><td>0.62</td><td>0.46</td><td>1.00</td><td>0.00</td><td>0.00</td><td>0.00</td><td>0.00</td><td>0.02</td></tr>
<tr><td>Retentate Split</td><td>0.38</td><td>0.54</td><td>0.00</td><td>1.00</td><td>1.00</td><td>1.00</td><td>1.00</td><td>0.98</td></tr>
</table>
</td>
<td>Vacuum Pressure (kPa)

Condenser Temperature (°C)

Component Split</td>
<td>3.47-5.47

-2 – 2

±10%</td>
</tr>
<tr>
<td>Liquid-liquid Extraction (Roffler et al., 1987)</td>
<td>


</td>
<td>Extractant: broth ratio

Preheater Temperature (°C)</td>
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80-100</td>
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</table>

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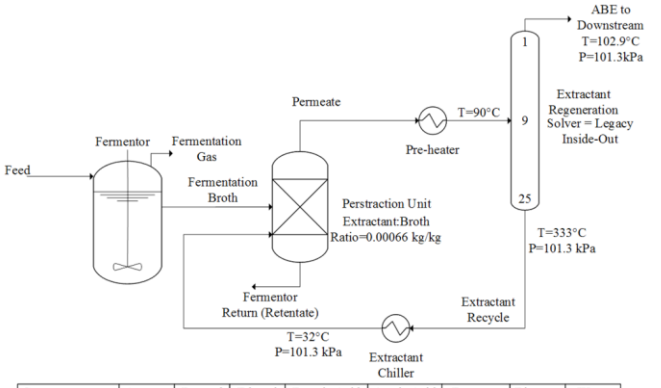
<td>Perstraction (Qureshi & Maddox, 2005)</td>
<td> 

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<tr><th>Component</th><th>Acetone</th><th>Butanol</th><th>Ethanol</th><th>Butyric Acid</th><th>Acetic Acid</th><th>Dextrose</th><th>Biomass</th><th>Water</th></tr>
<tr><td>Permeate Split</td><td>0.85</td><td>0.45</td><td>0.85</td><td>0.07</td><td>0.00</td><td>0.00</td><td>0.00</td><td>0.00</td></tr>
<tr><td>Retentate Split</td><td>0.15</td><td>0.55</td><td>0.15</td><td>0.93</td><td>1.00</td><td>1.00</td><td>1.00</td><td>1.00</td></tr>
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<td>Extractant: broth ratio

Preheater temperature (°C)

Component split</td>
<td>0.000594-0.000726

80-100

±10%</td>
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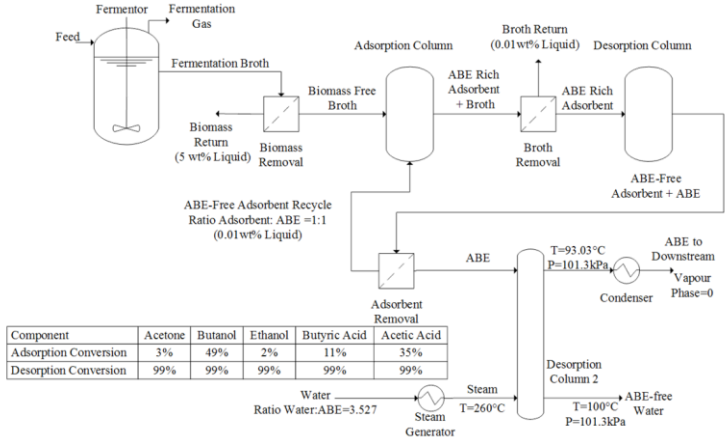
<tr>
<td>Adsorption (Yang et al., 1994)</td>
<td> 

<table>
<tr><th>Component</th><th>Acetone</th><th>Butanol</th><th>Ethanol</th><th>Butyric Acid</th><th>Acetic Acid</th></tr>
<tr><td>Adsorption Conversion</td><td>3%</td><td>49%</td><td>2%</td><td>11%</td><td>35%</td></tr>
<tr><td>Desorption Conversion</td><td>99%</td><td>99%</td><td>99%</td><td>99%</td><td>99%</td></tr>
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<td>Steam temperature (°C)

Adsorbent: broth ratio

Water: adsorbent ratio</td>
<td>250-270

0.9-1.1

3.177-3.877</td>
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Table 3: Economic assessment of the application of ISPR to an ABE production plant

			Batch	Flash	Vacuum	Gas Stripping	Pervaporation	LLE	Perstraction	Adsorption
Capital Cost		$ (million)	**10.1**	**68.4**	**54.7**	**68.4**	**68.4**	**54.7**	**54.7**	**54.7**
Annual Production	Acetone	kt/yr	3.9	10.7	10.7	10.7	10.7	10.7	10.7	10.7
	Butanol	kt/yr	13.7	37.5	37.5	37.5	37.5	37.5	37.5	37.5
	Ethanol	kt/yr	0.4	1.1	1.1	1.1	1.1	1.1	1.1	1.1
	TOTAL	kt/yr	18.0	49.3	49.3	49.3	49.3	49.3	49.3	49.3
Operational Cost	Feedstock Cost	$/yr (million)	9.6	26.4	26.4	26.4	26.4	26.4	26.4	26.4
	Energy Cost	$/yr (million)	1.4	8.1	5.0	11.5	4.8	7.4	3.8	4.5
	Other	$/yr (million)	2.3	6.3	6.3	6.3	6.3	6.3	6.3	6.3
	TOTAL	$/yr (million)	13.4	40.8	37.7	44.2	37.5	40.1	36.4	37.2
Product Sales	Acetone	$/yr (million)	4.5	12.5	12.5	12.5	12.5	12.5	12.5	12.5
	Ethanol	$/yr (million)	0.3	0.8	0.8	0.8	0.8	0.8	0.8	0.8
	Butanol	$/yr (million)	16.2	44.3	44.3	44.3	44.3	44.3	44.3	44.3
	Additional Products	$/yr (million)	4.1	11.3	11.3	11.3	11.3	11.3	11.3	11.3
	TOTAL	$/yr (million)	25.1	68.9	68.9	68.9	68.9	68.9	68.9	68.9
Profit		**$/yr (million)**	**11.8**	**28.1**	**31.2**	**24.7**	**31.3**	**28.8**	**32.4**	**31.6**

	Extra Capital	$ (million)		58.3	44.6	58.3	58.3	44.6	44.6	44.6
	Extra Profit	$/yr (million)		16.3	19.4	12.9	19.6	17.0	20.7	19.9
Additional Payback Time		**yr**		**3.6**	**2.3**	**4.5**	**3.0**	**2.6**	**2.2**	**2.2**
% Profit Increase				**139%**	**165%**	**110%**	**166%**	**145%**	**175%**	**169%**